

s7.m1.o5 In situ studies of zeolite dehydration

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Notes

Combining a heater, a powder diffractometer and an X-ray synchrotron source allows a wealth of not too fast reactions to be studied in situ. Not just to demonstrate a transition, but to determine and refine the crystal structures involved. The outcome is a detailed description of the transition including the chain of events leading up to it. The experimental set-up for such non-equilibrium studies are fairly simple, and there are several position sensitive detector systems available today^{1,7,8}. Dehydration processes in zeolites have in this way been successfully studied during recent years^{1,2,3,4,5,6}. Based on some 50 Rietveld refinements each, they have demonstrated that:

Water - Hydrogen bonding plays an important role in the charge compensation of the zeolite framework. The loss of water often triggers extra-framework cation migration to improve the charge compensation.

Extra-framework cations - The loss of water may also necessitate a reorganization of the cation coordination. In general the cations move closer to the framework to coordinate more framework oxygens. Further cation migration results in more effective use of the remaining water.

Framework - Zeolite frameworks are amazingly flexible. Variations in T-O-T-angles of 25° within 25 K are sometimes seen. Other zeolites respond by reconstructive framework rearrangements to maintain the cation coordination.

Phase transitions - The symmetry is in general limited by the framework topology and conformation. The symmetry is then reduced by the actual extra-framework cation and water arrangement. Thus, changes in the water and/or cation arrangement are more frequent causes for phase transitions than the more rare framework transformations.

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